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Transmitted Via Federal Express

April 9, 2003

Mr. Nabil Fayoumi
U.S. Environmental Protection Agency, Superfund Division
77 West Jackson Blvd (SR-6J)
Chicago, IL 60604

Re: Response to Second Round Comments on Quality Assurance/Sampling and Analysis Project Plan
BBL Project #: 10284.001 #2

Dear Mr. Fayoumi:

Enclosed is a comment/response package relating to the second round of comments on the draft Sauget Area 1 EE/CA-RI/FS Quality Assurance/Sampling and Analysis Project Plan (QA/SAPP). These comments were hand delivered by you during a meeting at the Solutia W. G. Krummrich Plant on February 25, 2003. The comments were not accompanied by a cover letter and, in consequence, no due date for the responses was established. Nevertheless, we have gone ahead and prepared the attached responses.

As we have done in the past, we are submitting only our responses to the comments, plan revisions, and specified plan components not included in the prior draft (not the revised QA/SAPP itself). When Solutia, Inc. and USEPA are agreed on the QA/SAPP revisions (after you have a chance to review the responses, revisions, and additions), we will modify the QA/SAPP and submit it to you as a final document.

Please note that we have addressed all of the second round review comments. Hopefully we will be able to proceed quickly to obtain approval to begin the sampling program. As we have indicated to you in the past, weather is rapidly becoming an important issue. If we miss the period when the Borrow Pit Lake is at low water level, we will need to wait until much later in this year (or possibly early next year) to complete the sampling.

If you have any questions, please contact Richard Williams of Solutia at 618-482-6340.

Sincerely,

BLASLAND, BOUCK & LEE, INC.

David F. Ludwig, Ph.D.
Vice President

DFL/krm
Enclosure

cc: Distribution

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TECHNICAL REPORT

Response to USPEA Second Round Comments on Quality Assurance/ Sampling and Analysis Project Plan

Sauget Area I
Dead Creek Sediment Removal Action
Mitigation Plan

Solutia, Inc.
Sauget, Illinois

April 2003

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Response to USEPA Second Round Comments on Quality Assurance/Sampling and Analysis Project Plan, Sauget Area 1, Dead Creek Sediment Removal Action Mitigation Plan

At Solutia's request, BBL Sciences has responded to comments received from the USEPA Region 5 on the Solutia-Sauget QA/SAPP revision dated January 30, 2003. The USEPA comments (in italics) and our responses to these comments (non-italics) are provided below for your consideration. As always, call us at 410-295-1205 at your convenience with comments, questions, or for additional information.

Section 4. Quality Objectives and Criteria for Measurement Data

This section should be included in QAPP Element A 7.

We will provide additional discussion of appropriate aspects of data quality objectives and measurement data criteria in QAPP Element 7. The USEPA Region 5 QAPP Instructions document specifies that these issues be addressed in detail in QAPP Element 4, so we will retain the full discussion here.

The Data Quality Objective (DQO) Process should include more project specific detail.

We will provide additional project-specific detail as appropriate regarding the DQO process (see specific discussions below).

The project should specify action levels, or threshold levels, for methyl mercury (MeHg) and mercury (Hg). Region 5 has Ecological Data Quality Levels (EDQLs) in Sediment and Soil for MeHg and Hg. The EDQL for MeHg in Sediment is 0.01 µg/kg and in Soil is 1.58 µg/kg. The Reporting Limit for MeHg in Sediments according to Table 3 is 0.0394 mg/kg, which is far greater than the EDQL of 0.01 µg/kg. The Region 5 EDQL for Hg (total) in Sediment is 174 µg/kg and in Soil is 100 µg/kg.

Perhaps, action levels were specified in the Dead Creek Sediment Removal Action Mitigation Plan (SRAMP). Perhaps, the USEPA Ecotox Thresholds are applicable. Illinois EPA must have data quality levels, too. Nevertheless, some values must be established to justify whether or not a Risk Assessment, Ecological or Human Health, should be performed.

One important objective of the sampling and analysis program is to provide data needed for risk assessment. This decision has already been made and agreed to by USEPA Region 5 and Solutia. Thus, no decision process or decision criteria are needed—the threshold decision regarding risk assessment has already been crossed. We will add text to the QAPP to reflect that Region 5 EDQLs (for methyl mercury and total mercury) and the total mercury sediment quality value from the Dead Creek SRAMP will be analytical quantitation goals. We have discussed these thresholds with the analytical laboratories, and they believe that these levels

can be achieved in many cases. We will provide additional text in the QAPP that will provide for the possibility that these levels can not be achieved in all samples. We will identify methods (statistical estimates and detection limit proportions) that will provide conservative input for risk assessment calculations for any samples in which the analytical limits exceed the EDQLs or SRAMP threshold.

In Step 4 describe the boundaries in more detail. Reference the maps for the sampling locations and specify the sampling depths. Discuss the temporal preferences, such as, sampling sediments without overlying water.

The revised QAPP will describe the boundaries in more detail, reference the maps regarding sampling locations, and specify sampling depths. We will discuss the preference (indeed, requirement) for sampling without (or with minimal) overlying water.

In Step 5 describe the decisions rules in more detail. Again, there must be some action levels that will require a risk assessment, further investigative sampling, sampling at greater depths, remediation, removal, or other courses of action.

As discussed previously, no decision rules are needed because it has already been acknowledged and agreed by USEPA Region 5 and Solutia that a risk assessment will be conducted. We will add text to discuss the role of the data in the risk assessment and the appropriateness of the analytical quantitation goals.

In Step 6 define the 2 types of decision errors and specify limits on decision errors. There must be some project action or threshold levels. Results above action levels will require risk assessment or further investigation. If any results are below action levels, discuss the possible decision errors for terminating the investigation. A Decision Performance Goal Diagram could be constructed to assist in the decisions. Reference the specific sections and tables of the QAPP that are applicable.

As discussed previously, decision error limits will not affect the outcome of the project—a risk assessment application has already been agreed to by USEPA Region 5 and Solutia. We will add text discussing the effect of analytical uncertainty on risk assessment conclusions (i.e., how analytical uncertainty will be accommodated in the Uncertainty Analysis section of the Risk Assessment Report).

In Step 7 choose statistical tests and computer modeling to help optimize sample design. If further investigative sampling is necessary, the DQOs and the sampling design could be refined.

Sample design has already been agreed to by USEPA Region 5 and Solutia. We will add text discussing the level of resolution provided by the agreed-upon sample design, and its appropriateness for risk assessment (i.e., how the sample design contributes to minimal uncertainty in risk assessment conclusions and thus in subsequent risk management decisions).

SOP #BR-0011 Determination of Methyl Mercury by Aqueous Phase Ethylation, etc.
Brooks Rand, LLC

A. *This method is a modification and deviation from the Method 1630. Provide method performance data demonstrating the method achieves acceptable accuracy and precision. Example chromatograms should be included.*

USEPA Method 1630 is a draft method that has not undergone review or method validation by the USEPA and is written for water samples. Comparison of actual lab performance for methyl mercury in sediments to the draft criteria for water samples may not be appropriate. The revised Standard Operating Procedure (SOP) will include actual lab performance QA control charts and example chromatograms as attached to this Memo.

B. Section 2
Provide the method detection limit (MDL) and reporting limit (RL), or practical quantitation limit (PQL), for Mono-Methylmercury (MMHg).

The method detection limit (MDL) and practical quantitation limit (PQL) will be provided in the revised SOP. The MDL based on the results of a formal MDL study is 0.02 ng/g and the method limit (ML; equivalent to PQL) is 0.07 ng/g (ng of Hg existing as monomethylmercury per gram of wet sediment sample). Based on more recent method blank data and low level standards an MDL of 0.003–0.005 ng/g and an ML of 0.01 ng/g are achievable in many cases.

C. Section 7.2.2
Describe the composition and number of method blanks.
Describe the preparation and concentration of the MS/MSDs.

The revised SOP will describe the composition and number of method blanks as follows: Method blanks are prepared and analyzed as samples except without the addition of any sample. Method blanks consist of the same quantities of the same reagents used to prepare samples. Three method blanks are prepared and analyzed with each batch of samples.

The revised SOP will describe the preparation and concentration of the matrix spikes/matrix spike duplicates (MS/MSDs) as follows: MS/MSDs are prepared in the same manner as native samples except that they have a known quantity of monomethylmercury standard added to the sample aliquots prior to preservation. Samples are typically spiked with 1 ng of monomethylmercury per gram of sample. Spiking levels can be adjusted based on project-specific requirements or on expected concentration ranges for the samples used for MS/MSD purposes.

D. Section 9
Denote if results will be reported on a wet or dry weight basis.

Sample results for monomethylmercury in sediments are reported as specified in the project-specific requirements. When percent solids determinations are requested as one of the

analytical parameters to be performed by Brooks Rand, then final sample results are reported on a dry weight basis. Raw data is typically reported on a wet weight basis.

E. Section 10
Discuss corrective actions if quality control (QC) acceptance criteria given on page BR- 0011-18 are not achieved

Text will be added to the SOP discussing corrective actions if QC acceptance criteria are not achieved.

F. Section 10.2, typo
Clarify reference to "section 8.4", since this section is not present.

This reference will be changed to read 'section 10.4' in the revised SOP.

G. Section 10.6
The Method 1630 recommends 3 method blanks. Moreover, the Method 1630 recommends Ethylation Blanks.

The SOP will be revised to indicate that three method blanks will be prepared and analyzed with each batch of samples. The ethylation blanks referred to in Method 1630 are the same as the calibration blanks and calibration blank checks performed in SOP BR-0011. Four ethylation blanks are analyzed prior to instrument calibration and then additional ethylation blanks are analyzed following each Ongoing Precision and Recovery (OPR) sample. Additionally ethylation blanks are analyzed immediately following the analysis of any sample that is greater than twice the level of the highest standard using the same bubbler and trap to check for contamination.

H. Section 10.7
The Method 1630 indicates that if the RPD exceeds QC criteria, the analytical batch should be reanalyzed. Also, if field duplicates exceed RPD QC criteria, the field sampling team should be notified to initiate corrective actions.

Text will be added to the revised SOP to indicate that if the relative percent difference (RPD) exceeds QC criteria, the analytical batch is reanalyzed or re-prepared and reanalyzed as necessary. Text will also be added to the revised SOP to indicate that if the sampling team has clearly identified field duplicates as such, then the field sampling team is contacted when field duplicates fail to meet the QC criteria for RPD and notified to initiate corrective actions.

Battelle Marine Sciences Laboratory Standard Operating Procedure (SOP) MSL-I-016-05

A. Sections 1.0 and 4.1
This method is a modification and deviation from the EPA Method 245.5 and SW-846 7471A, since Potassium Permanganate is not included in the digestion. Would this digestion procedure be applicable for these sediment samples, which may contain high concentrations of Mercury and

Organomercury compounds? Provide method performance data demonstrating the method achieves acceptable accuracy and precision.

The method described in this SOP is used by the National Oceanic and Atmospheric Administration (NOAA) National Status and Trends (NS&T) program for the analysis of bottom sediment collected as part of the Mussel Watch Project, thus it is appropriate for sediment samples that may contain high concentrations of mercury and organomercury compounds (NOAA, 1998).

An extensive quality control/quality assurance program is part of the NOAA NS&T program to ensure that data are comparable between all participating laboratories (including Battelle Marine Sciences Laboratory). All NS&T laboratories are required to participate in a continuing series of inter-comparison exercise utilizing a variety of solutions and natural matrix materials, with the results published as a number of NOAA technical memoranda (NOAA, 1992; 1993; 1995a,b,c,d,e). These reports include method performance data for all NS&T methods, as well as the method used in the QA/SAPP for total mercury. The revised SOP will include discussion of these reports and the appropriateness of the methods for the Borrow Pit Lake investigation.

B. Section 4.4
Specify the sample amount and the concentration of the matrix spike.

The range of acceptable sample volumes is specified in Section 4.4.4 of the SOP, which reads "...there should be at least 6 ml of sample in the test tube, which does not hold more than 9 ml...3 ml of digestate and 3 ml of 3% HNO_3 or 1 ml of digestate and 5 ml of 3% HNO_3 are the common dilutions used currently."

The concentration of the matrix spike cannot be indicated in the SOP because the actual concentration can vary considerably based on the concentration of the representative sample used in the MS. A representative sample is used as a MS to check for matrix interference.

C. Section 5 and Table 1
Include calculations for determining % Recovery and RPD.

Calculations for determining % Recovery and RPD will be included in the Revised QA/SAPP.

D. Section 6.1
Denote the composition of the method blank and concentration of the matrix spike.

The composition of the method blank is specified in Section 6.1 as follows "A method blank consists of all reagents used in the digestion procedure and it is digested and analyzed as a sample." The concentration of the MS cannot be indicated in the SOP because the actual concentration can vary considerably based on the concentration of the representative sample used in the MS. A representative sample is used as a MS to check for matrix interference.

Literature Cited

NOAA. 1992. *Elemental analyses in marine sediment and biological tissues.* NOAA Tech. Memo NOAA/NOS/ORCA 66. National Oceanic and Atmospheric Administration, Rockville, MD.

NOAA. 1993. *Quality Assurance Project trace organic inter-comparison exercise results 1986-1990.* NOAA Tech. Memo NOAA/NOA/ORCA 69. National Oceanic and Atmospheric Administration, Silver Spring, MD.

NOAA. 1995a. *Quality Assurance Project inter-comparison exercise results 1991-1993.* NOAA Tech. Memo NOAA/NOS/ORCA 79. National Oceanic and Atmospheric Administration, Silver Spring, MD.

NOAA. 1995b. *NOAA Status and Trends Program sixth round inter-comparison exercise results for trace metals in marine sediments and biological tissues.* NOAA Tech. Memo NOAA/NOS/ORCA 85. National Oceanic and Atmospheric Administration, Silver Spring, MD.

NOAA. 1995c. *NOAA Status and Trends Program seventh round inter-comparison exercise results for trace metals in marine sediments and biological tissues.* NOAA Tech. Memo NOAA/NOS/ORCA 84. National Oceanic and Atmospheric Administration, Silver Spring, MD.

NOAA. 1995d. *NOAA Status and Trends Program eighth round inter-comparison exercise results for trace metals in marine sediments and biological tissues.* NOAA Tech. Memo NOAA/NOS/ORCA 83. National Oceanic and Atmospheric Administration, Silver Spring, MD.

NOAA. 1995e. *NOAA Status and Trends Program eighth round inter-comparison exercise results for trace metals in marine sediments and biological tissues.* NOAA Tech. Memo NOAA/NOS/ORCA 93. National Oceanic and Atmospheric Administration, Silver Spring, MD.

NOAA. 1998. *A summary of data on tissue contamination from the first three years (1986-1988) of the mussel watch project.* NOAA Tech. Memo NOS OMA 49. National Oceanic and Atmospheric Administration, Rockville, MD.